luminescence measurement technique is to be preferred over indirect methods even when the latter, as here, have been carried out with the greatest care. Currently we are setting up for time-resolved infrared luminescence measurements in order to confirm our current indications that τ_Δ in C_6D_6, CD_3CN, and $(CD_3)_2CO$ is close to 1000 μs as shown[11] for $CDCl_3$. On the other hand, we contend that for τ_Δ values of less than ca. 200 μs, careful use of the time-resolved substrate bleaching method following flash excitation gives very reliable results.

Acknowledgment. Work at the Center for Fast Kinetics Research is supported by the Biotechnology Branch of the Research Resources Division of NIH (RR00886) and by the University of Texas. Partial support for this work comes from NIH Grant No. GM 24235.

Synthesis, Structure, and Electrochemical Behavior of *cis*-Diammineplatinum(III) Dimers with Bridging α-Pyridonate Ligands

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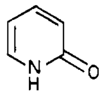
cis-Diammineplatinum α-pyridone blue, $[Pt_2(NH_3)_4(C_5H_4NO)_2]_2(NO_3)_5\cdot H_2O$ (PPB), **1**, is a tetranuclear, mixed-valent, crystalline compound comprised formally of three Pt(II) and one Pt(III) centers linked by bridging α-pyridonate ligands, partial metal–metal bonding, and interplanar N—H···O hydrogen bonding.[1] In solution the compound is unstable, as determined by the loss of its blue color and paramagnetism with time.[2] Recently we have been studying further the reactions of α-pyridone with complexes of *cis*-diammineplatinum(II)[3] in order to provide additional clarification of the chemical and electronic spectral properties of PPB. Here we show that PPB can be oxidized to a metal–metal bonded platinum(III) dimer of which there are few well-characterized examples in the literature.[4] The crystal structure of the red-orange product reveals it to be the head-to-head isomer of *cis*-$[(H_2O)(NH_3)_2Pt(C_5H_4NO)_2Pt(NH_3)_2(NO_3)](NO_3)_3\cdot 2H_2O$ (**2**). We also report the synthesis of the head-to-tail isomer, and electrochemical studies that reveal a quasi-reversible two-electron redox process for the $[Pt(II)]_2/[Pt(III)]_2$ couple. Concerted two-electron transfer reactions of this kind are rare for binuclear transition-metal complexes[5] and therefore of considerable interest.



α-pyridone

Red crystals of **2** were obtained by heating 20 mg of **1** to 80 °C in 5 mL of 5 M nitric acid for 2 min during which time the solution changed color from blue to red orange. Air evaporation of the resulting solution provided a 65–80% yield of **2**. The head-to-tail isomer, *cis*-$[(NO_3)(NH_3)_2Pt(C_5H_4NO)_2Pt(NH_3)_2(NO_3)](NO_3)_2\cdot H_2O$ (**3**), was prepared in a similar fashion from the head-to-tail platinum(II) dimer $[Pt(NH_3)_2(C_5H_4NO)]_2(NO_3)_2\cdot 2H_2O$ (**4**)[3] and isolated as red crystals. In addition to a complete X-ray crystal structure analysis of compound **2**,[6] both

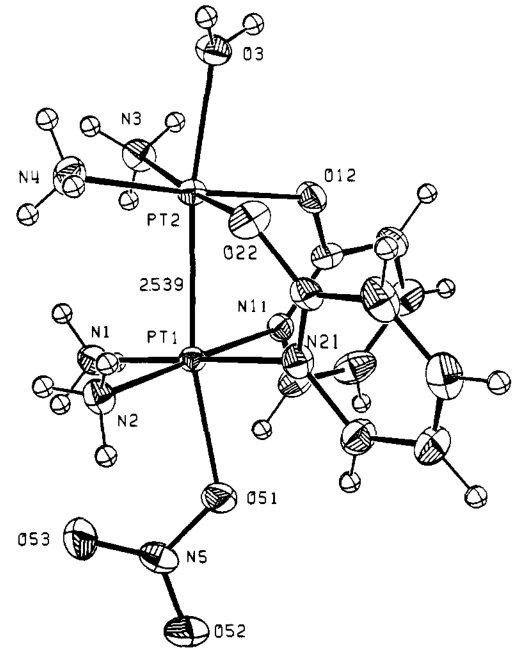


Figure 1. ORTEP drawing of the structure of the head-to-head isomer of the *cis*-$[(H_2O)(NH_3)_2Pt(C_5H_4NO)_2Pt(NH_3)_2(NO_3)]^{3+}$ cation in compound **2**, showing the 40% probability thermal ellipsoids. For clarity, hydrogen atoms are depicted as spheres with B set arbitrarily to 1 Å^2. The nitrate oxygen O(53) is intramolecularly hydrogen bonded to the ammine ligands (N1 and N2) while the coordinated H_2O (O3) is hydrogen bonded to a lattice water molecule.

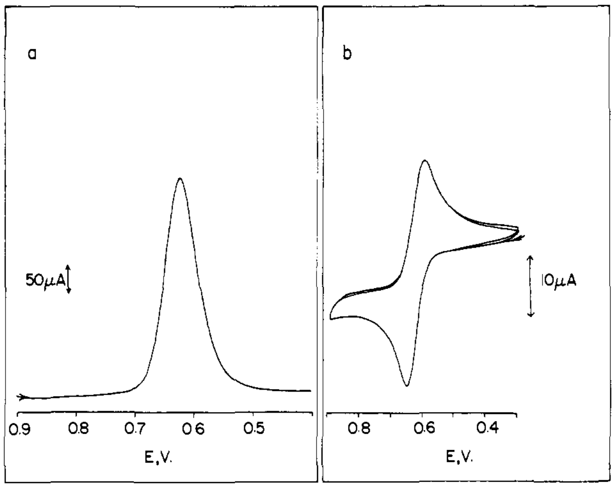


Figure 2. (a) Differential pulse voltammogram (Δi vs. V) of compound **3**, 0.4 mM. (b) Cyclic voltammogram for compound **4** (several cycles obtained at a scan rate of 20 mV s^{-1}), 0.9 mM. Experimental details are given in ref 8.

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(6) X-ray analysis: Compound **2** crystallizes in the monoclinic system (space group $P2_1/n$) with the following cell parameters: $a = 9.709$ (1), $b = 13.779$ (2), $c = 18.655$ (2) Å; $\beta = 94.44$ (1)°; $\rho_{obsd} = 2.50$ (2), $\rho_{calcd} = 2.51$ g/cm^3; $Z = 4$. The structure was solved by standard Patterson and Fourier methods using 5715 unique reflections collected out to $2\theta = 55°$ on a Nonius CAD-4F diffractometer with Mo Kα ($\lambda = 0.7017$ Å) radiation. Refinement of the absorption corrected data with all atoms assigned anisotropic temperature parameters, except hydrogens which were refined isotropically with constraints, converged to a value of 0.032 for the discrepancy index $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$. Atomic positional and thermal parameters are provided as supplementary material. Full details will be reported at a later date.

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Table I. Geometric Properties of Related Platinum Complexes

compound	formal Pt oxdn state	distance, Å: Pt–Pt	Pt–NH_3	Pt–N	Pt–O	Pt–L_{axial}	dihedral angle, deg[a]: τ	ω	ref
$[Pt_2(NH_3)_4(C_5H_4NO)_2]_2(NO_3)_5 \cdot H_2O$ (1)	2.25	2.775 (1) 2.877 (1)	2.06 (av)	2.06 (av)	2.04 (av)	3.32 (NO_3^-)	27.4	22	1
$[Pt_2(NH_3)_4(C_5H_4NO)_2(NO_3)(H_2O)](NO_3)_3 \cdot 2H_2O$ (2)	3.0	2.539 (1)	2.05 (av)	2.06 (av)	2.00 (av)	2.11 (H_2O) 2.18 (NO_3^-)	20.2	23	*b*
$[Pt_2(NH_3)_4(C_5H_4NO)_2](NO_3)_2 \cdot H_2O$ (4)	2.0	2.898 (1)	2.05 (av)	2.03	2.02		28.9	13	3
$[Pt_2(NH_3)_4(C_5H_4NO)_2]_2(NO_3)_4$ (5)	2.0	2.877 (1) 3.129 (1)	2.05 (av)	2.05 (av)	2.04 (av)		30.0	21	3
$[Pt_2(CH_3)_4(C_2F_3O_2)_2(C_6H_7N)_2]$ (6)	3.0	2.557 (1)	2.03 (av)[c]		2.19 (av)	2.09 (MePy) 2.17 (MePy)	*d*	22	4[c]
$K_2[Pt_2(SO_4)_4(H_2O)_2]$ (7)	3.0	2.466 (d)			1.99 (av)	2.12 (H_2O)	*d*	*d*	4[a]
$(H_5O_2)[Pt_2(NH_3)_4(C_5H_6N_3O)_2(NO_2)_2](NO_3)_2$ (8)	2.5[e]	2.584 (1)	2.07 (av)	2.03 (av)	2.01 (av)	2.12 (NO_2^-) 2.13 (NO_2^-)	21	25	11

[a] τ is the tilt angle between adjacent platinum coordination planes and ω is the average torsion (or twist) angle about the Pt–Pt vector. [b] This work. [c] Pt–C distance. [d] Values not reported. [e] See text.

Table II. Electrochemical Data[a]

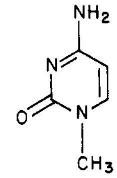
compd	CV: ΔE_{pp}[b]	$E_p - E_{p/2}$[c]	$\Delta E_{1/2}$[d]	$E^1_{1/2}$	$E^2_{1/2}$	DPV: width[e]	$\Delta E_{1/2}$[d]	$E^1_{1/2}$	$E^2_{1/2}$	CPC: no. of electrons
2	80(10)	52(8)	30(10)	0.63(1)	0.60(1)	110(5)	50(10)	0.65(1)	0.60(1)	1.8
4	45(10)	35(4)	−30(20)	0.61(1)	0.64(1)	60(5)	−20(10)	0.61(1)	0.63(1)	1.9

[a] All reported potentials are in V vs. SCE; see ref 8 for experimental details. [b] Peak-to-peak separation, $\Delta E_{pp} = |E_p(\text{cathodic}) - E_p(\text{anodic})|$, in mV. [c] Width at $i_d/2$, in mV. [d] $E^1_{1/2} - E^2_{1/2}$, in mV. [e] Full width at half-i_{max_3} in mV.

2 and **3** have been characterized by analytical,[7] spectroscopic,[7] and electrochemical[8] data.

The molecular structure of the $[(H_2O)(NH_3)_2Pt(C_5H_4NO)_2Pt(NH_3)_2(NO_3)]^{3+}$ cation is illustrated in Figure 1. Table I contains a comparison of the geometry of this complex with those of previously reported platinum(III) compounds and the relevant *cis*-diammineplatinum(II) complexes of α-pyridone. The Pt–Pt distance in **2** is 2.539 (1) Å, with both platinum(III) atoms being six-coordinate. This bond distance compares favorably with the 2.557 (1) Å distance reported for the structurally analogous trifluoroacetate-bridged platinum(III) dimer $[(MePy)(CH_3)_2Pt(O_2CCF_3)_2Pt(CH_3)_2(MePy)]$ (**6**); MePy = 4-methylpyridine.[4c] It is somewhat longer than the value of 2.466 Å reported for the sulfate-bridged dimer $K_2[Pt_2(SO_4)_4(H_2O)_2]$ (**7**).[4a] As with some of the multiply metal–metal bonded complexes of the early transition metals,[9a,b] the Pt(III) dimers **2**, **6**, and **7** contain tightly bound axial ligands that are capable of influencing the metal–metal bond distance.[9c,10] Thus the somewhat longer Pt–Pt distance in **6** compared with **2** may be due in part to the better donor ability of MePy compared to water and nitrate ion. In the case of the platinum(III) sulfate dimer **7**, both the presence of weak axial donors (H_2O) and the lack of nonbonded repulsions between the in-plane ligands, NH_3 in **2** and CH_3 in **6**, contribute to the very short observed metal–metal distance. In all three Pt(III) dimers there is a Pt–Pt single bond as expected in a d^7–d^7 system with the electronic configuration $\sigma^2\pi^4\delta^2\delta^{*2}\pi^{*4}$,[9a,b] or a slightly modified version thereof.[9c]

The similarity between the structures of the *cis*-diammineplatinum(III) α-pyridonate bridged dimer **2** and the oxidized head-to-tail 1-methylcytosine dimer $(H_5O_2)[(NO_2)(NH_3)_2Pt(C_5H_6N_3O)_2Pt(NH_3)_2(NO_2)](NO_3)_2$ (**8**)[11] is striking. Compound **8** has a Pt–Pt distance of 2.584 (1) Å, two axially bonded nitrite ligands, two bridging 1-methylcytosine ligands linked to platinum through N(3), and a deprotonated exocyclic amine (4-NH_2) group. From the X-ray structure of compound **8** the oxidation state of the platinum atoms in the dimer was assigned as +2.5. This conclusion was based on charge considerations resulting from the identification of the $H_5O_2^+$ cation through a short O···O distance of 2.50 (4) Å found in the crystal structure and on the value of the interatomic Pt–Pt distance.[11] While this formulation may be correct, the Pt–Pt bond length is only 0.045 Å longer than found for **2**. Since the structural trans effect of $NO_2^- > MePy > NO_3^- \geq H_2O$ would be expected to lengthen the Pt–Pt distance in **8** vs. **2** or **6**,[10] it is possible that **8** is actually a platinum(III) dimer. Further studies of **8** should be performed to verify the assigned oxidation state.



1-methylcytosine

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[7] Chemical and spectroscopic data. Anal. Calcd for $Pt_2C_{10}H_{22}N_{10}O_{15}$, the dehydrated form (dried sample) of **2**: C, 13.16; H, 2.43; N, 15.35. Found: C, 13.16; H, 2.45; N, 15.17. UV-visible spectrum of **2** in water: λ 254 (ϵ 26 000 cm^{-1} M^{-1}), 268 (sh), 298 (sh), 335 (sh), 408 (sh) nm. Anal. Calcd for $Pt_2C_{10}H_{20}N_{10}O_{14}$, the dehydrated form (dried sample) of **3**: C, 13.43; H, 2.25; N, 15.66. Found: C, 13.34; H, 2.25; N, 15.56. UV-visible spectrum of **2** in water: λ 250 (ϵ 25 000 cm^{-} M^{-1}), 275 (sh), 305 (sh), 340 (sh), 420 (sh) nm. Both **2** and **3** exhibited no ESR signal in the solid state, confirming their diamagnetism.

[8] Electrochemical methods: Cyclic voltammetry (CV) and controlled potential coulometry (CPC) experiments were performed with a PAR Model 173 potentiostat equipped with a Model 179 digital coulometer and a PAR Model 175 universal programmer. Differential pulse voltammetry (DPV) was carried out with a PAR Model 174A analyzer. All CV and DPV experiments were performed in 1 M KNO_3 (adjusted to pH 1 with nitric acid) by using a three electrode system consisting of a platinum bead working electrode, a platinum wire loop auxiliary electrode, and a PAR SCE reference electrode which was separated from the working solution by a Vycor plug. CPC experiments were carried out under identical conditions in a standard H cell by using a platinum wire loop working electrode. All DPV measurements were made at a sweep rate of 2 mV s^{-1} with a pulse height of 10 mV and a "drop time" of 0.5 s.

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[10] We have recently confirmed this expectation by X-ray structural studies of the head-to-tail dimers $[X(NH_3)_2Pt(C_5H_4NO)_2Pt(NH_3)_2X](NO_3)_2 \cdot H_2O$, X = NO_2 and NO_3, and will report on them shortly.

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J. Am. Chem. Soc. **1981**, *103*, 6763–6765 6763

The results of the electrochemical studies of compounds **2** and **4** are summarized in Table II. Typical differential pulse and cyclic voltammograms are shown in Figure 2. Electrochemical oxidation of the head-to-tail platinum(II) dimer (**4**) takes place reversibly at scan rates (v) slower than 50 mV s^{-1} with only one wave observed at $E_p = +0.63$ V in the range examined, from +1.0 to −0.1 V, with CV. At scan rates faster than 50 mV s^{-1} both ΔE_{pp} and the anodic to cathodic current ratio (i_a/i_c) increase, and $i_a v^{-1/2}$ decreases as v increases. These observations are consistent with a chemical reaction coupled with oxidation of the Pt(II) complex.[12] This reaction may involve coordination of axial ligands (NO_3^- or H_2O) to the resulting Pt(III) dimer. Exhaustive electrolysis of **4** at +0.85 V produced a colorless to orange color change in the solution and a net loss of 0.95 electrons per platinum atom. CV experiments conducted after the electrolysis or on authentic samples of **3** gave results identical with those obtained on **4** prior to electrolysis. From the CV data obtained on **3** and **4** at slow scan rates the difference in the reduction potentials, $\Delta E_{1/2} = E^1{}_{1/2} - E^2{}_{1/2}$, was estimated to be −30 mV.[5c,13] This value is in reasonable agreement with the value of −20 mV obtained for $\Delta E_{1/2}$

$$\mathrm{Pt^{III}Pt^{III}} + e^- \overset{E^1{}_{1/2}}{\rightleftharpoons} \mathrm{Pt^{II}Pt^{III}} + e^- \overset{E^2{}_{1/2}}{\rightleftharpoons} \mathrm{Pt^{II}Pt^{II}}$$

from the DPV experiment (Table II). The negative value obtained for $\Delta E_{1/2}$ indicates that, in the oxidation of **4**, the removal of the second electron is less difficult than the first. This result appears to be one of the few reported such cases in inorganic systems.[5]

The electrochemical behavior of the head-to-head Pt(III) dimer **2** is similar in some respects to that observed for the head-to-tail isomer (Table II). A single wave was observed at $E_p = +0.63$ V in both CV and DPV studies, and the redox process also appeared to involve a coupled chemical reaction following an overall two-electron transfer. The major difference found was in the kinetics of the coupled chemical reaction. Although this system approaches quasi-reversible behavior at slow scan rates, ΔE_p, i_a/i_c, and $i_p v^{-1/2}$ all vary as a function of v down to the slowest measured scan rate (5 mV s^{-1}). Controlled potential electrolysis of **2** showed that an additional chemical reaction occurs after the electron transfer. As the reductive electrolysis proceeded to completion, the solution changed color from orange to green to blue and finally became colorless. The blue color produced during this reversible process is almost certainly due to the formation of the mixed-valence PPB, **1**. A blue color does not form in the controlled potential electrolysis of **4** presumably because the head-to-tail isomer cannot associate into a tetranuclear species, a result of steric effects which prevent the close approach of two dimeric units. If one assumes that the CV and DPV data for **2** approach reversible behavior in the slow scan rate limit, approximate values of +30 to +50 mV can be obtained for $\Delta E_{1/2}$ (Table II). This result implies that, for the head-to-head isomer, the second electron is harder to remove than the first. The mixed valence Pt(II)–Pt(III) dimer is therefore a possible intermediate in the electrochemical formation of PPB. In particular, reaction of the Pt(II)–Pt(III) intermediate with a Pt(II)–Pt(II) species, formed by disproportionation of the mixed-valence dimer, would generate the tetranuclear blue complex, PPB, having the formal oxidation state of +2.25. Further chemical and electrochemical studies of this system are in progress.

Acknowledgment. This work was supported by National Institutes of Health Research Grant CA-15826. We thank Engelhard Industries for a loan of K_2PtCl_4 used to make all platinum complexes described.

Supplementary Material Available: Atomic positional and thermal parameters for compound **2** (2 pages). Ordering information is given on any current masthead page.

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Biosynthesis of Dibromotyrosine-Derived Antimicrobial Compounds by the Marine Sponge *Aplysina fistularis* (*Verongia aurea*)[1,2]

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Marine natural products have been a focus of recent interest as potential pharmaceuticals, with sponges proving to be particularly rich in bioactive compounds. The brominated phenols and quinones isolated from *Aplysina* (*Verongia*) species include active antibacterial agents which are proposed to be derived from mono- or dibromotyrosines.[3] Attempts by other workers to demonstrate such a conversion in vivo have been unsuccessful.[4,5] However, we have now reinvestigated the biosynthesis of brominated phenols and bromoquinones in *Aplysina fistularis* by using liposome-enclosed precursors and have demonstrated the conversion of phenylalanine (Phe) and tyrosine (Tyr) to the dienone **3** as well as to the rearranged product dibromohomogentisamide (**4**). This work supports the biosynthetic pathway in Scheme I.

Samples of *Aplysina fistularis*, with their natural rock substrates intact, were collected by scuba operations at several coves offshore from the Catalina Marine Science Center, Santa Catalina Island, CA. An antibiotic dip of benzylpenicillin, streptomycin sulfate, and chloramphenicol in sea water was used to prevent the bacterial infection normally observed after the organisms are handled.[8] Sponges were incubated in 3 L of aerated sea water in polyethylene containers maintained at environmental temperatures by a fresh running sea water bath. Algal growth was inhibited by limiting the light incident on the aquaria. These precautions allowed sponge survival in the laboratory for as long as 2 weeks.[9]

Conventionally, radiolabeled precursors have been administered to sponges by simple solution in sea water.[4,6,7] Since sponges are known to filter feed on particulates such as dead bacteria,[10] we presumed that liposome encapsulation would improve incorporation of precursors by sponges. Multilamellar lipid vesicles of bacterial dimensions were prepared by agitating an aqueous solution of the precursor in a flask coated with a lipid film.[11] Phosphatidylcholine

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[1] Presented at the Marine Chemistry Symposium, 64th Conference, Chemical Institute of Canada, Halifax, Nova Scotia, June 4, 1981.

[2] Dr. Robert Given, Catalina Marine Science Center, originally identified the bright yellow sponge as *Verongia aurea sensu* de Laubenfels, 1948. This sponge has been reclassified as *Aplysina fistularis* (Pallas) *sensu* Wiedenmayer, 1977 (Dr. G. J. Bakus, University of Southern California, personal communication).

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[9] Survival of the sponges was clearly dependent on the sea water temperature and dropped off rapidly above 17 °C.

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